



UNIVERSITI PUTRA MALAYSIA

**SYNTHESIS AND CHARACTERISATION OF
THERMOTROPIC LIQUID CRYSTALLINE MONOMER:
4-METHYLPHENYL 4- [2-(VINILOXY)ETHOXY]BENZOATE**

MOHAMAD HUSSIN BIN HAJI ZAIN

FSAS 2000 18

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4-METHYLPHENYL 4-[2-(VINILOXY)ETHOXY]BENZOATE**

By

MOHAMAD HUSSIN BIN HAJI ZAIN

**Thesis Submitted in Fulfilment of the Requirement of the Degree of Master
of Science in the Faculty of Science and Environmental Studies
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December 2000



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science.

SYNTHESIS AND CHARACTERISATION OF THERMOTROPIC LIQUID CRYSTALLINE MONOMER: 4-METHYLPHENYL 4-[2-(VINILOXY)ETHOXY]BENZOATE

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Faculty: Science and Environmental Studies

This thesis is primarily concerned with the synthesis of a new thermotropic liquid crystalline monomer and its characterisation by using elemental analysis, spectroscopic analysis and thermal analysis techniques.

A new thermotropic liquid crystalline monomer, 4-methylphenyl 4-[2-(viniloxy)ethoxy]benzoate (4MP4VEB) was synthesised by reacting 4-methylphenyl 4-hydroxybenzoate (4MP4HB) with 2-chloro-ethyl-vinyl ether in acetonic solution. The compound 4MP4HB as a mesogenic group was prepared from the reaction of *p*-hydroxybenzoic acid and *p*-cresol. The mesogenic group (4MP4HB) was also used to synthesise the other thermotropic liquid crystalline compound namely 4-methylphenyl 4-(3-hydroxypropoxy)benzoate (4MP4HPB) but without a monomer terminal.

The attempted synthesis of other liquid crystalline monomer with a varying spacer length using a different approach only ended up with a precursor compound. Two precursors, namely 4-[4-(allyloxy)butoxy]benzoic acid (4ABBA) and 4-(allyloxy)benzoic acid (4ABA) were successfully synthesised for the preparation of thermotropic liquid crystals with an allyl group as a monomer terminal. The compound 4ABBA is a precursor that has a spacer linkage, while 4ABA is without a spacer linkage.

The liquid crystalline compound and the synthesised precursors were characterised by elemental, spectroscopic and thermal analyses. The observation of thermal properties and liquid crystallinities under polarising microscope revealed that the compound 4MP4VEB exhibited the like-mosaic defect texture of nematic mesophase, while the compound 4MP4HPB exhibited the focal-conic texture of smectic A mesophase.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains.

**PENYEDIAAN DAN PENCIRIAN MONOMER HABLUR
CECAIR BERTERMOTROPIK: 4-METHYLPHENYL 4-[2-
(VINYLOXY)ETHOXY]BENZOATE**

Oleh

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Tesis ini mengutamakan penumpuan terhadap penyediaan monomer hablur cecair bertermotropik dan penciriannya dengan menggunakan teknik analisis kandungan unsur, spektroskopi dan termal.

4-metilfenil 4[2-(viniloksi)etoksi]benzoat (4MP4VEB) ialah monomer termotropik hablur cecair yang telah disintesiskan dari tindakbalas antara 4-metilfenil 4-hidroksibenzoat (4MP4HB) dengan 2-kloro-etil-vinil eter di dalam larutan beraseton. Sebatian 4MP4HB adalah kumpulan mesogen yang telah disediakan dari tindakbalas antara asid *p*-hidrosilbenzoik dan *p*-kresol. Sebatian mesogen ini juga digunakan untuk menyediakan satu lagi sebatian termotropik hablur cecair yang tidak mempunyai monomer penghujung iaitu 4-metilfenil 4-(3-hidroksipropoksi)benzoat (4MP4HPB).

Suatu pendekatan lain yang dilakukan untuk mensintesis monomer hablur cecair bertermotropik yang mempunyai pelbagai panjang kumpulan peruang hanya berjaya setakat penghasilan sebatian perantaraan sahaja. Asid 4-[4-(aliloksi)butoksi]benzoik (4ABBA) dan asid 4-(aliloksi)benzoik (4ABA) adalah dua sebatian perantaraan yang mempunyai kumpulan alil sebagai monomer penghujung yang telah berjaya disediakan. Sebatian 4ABBA ialah sebatian perantaraan yang mempunyai kumpulan peruang antara monomer penghujung dan kumpulan mesogen tetapi sebatian 4ABA mempunyai kedudukan sebaliknya.

Sebatian berhablur cecair dan sebatian perantaraan ini telah dilakukan pencirian dengan menganalisa kandungan unsur, spektroskopi dan termalnya. Pemerhatian terhadap sifat-sifat termal dan kehablurcairan dengan menggunakan mikroskop berkutub menunjukkan yang sebatian 4MP4VEB telah menghasilkan rupa-bentuk seperti kecacatan mozek pada fasa-meso nematik dan sebatian 4MP4HPB pula menghasilkan rupa-bentuk fokal-berkon pada fasa-meso smektik A.

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I certify that an Examination Committee met on 12th December 2000 to conduct the final examination of Mohamad Hussin Bin Haji Zain on his Master of Science thesis entitled "Synthesis and Characterisation of Thermotropic Liquid Crystalline Monomer: 4-methylphenyl 4-[2-(vinylloxy)ethoxy]benzoate" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulation 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:


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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

Signed



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Date: 09th February 2001

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LIST OF ABBREVIATIONS

LC	liquid crystal
LLCs	lyotropic liquid crystals
TLCs	thermotropic liquid crystals
LCPs	liquid crystal polymers
SCLCPs	side chain liquid crystal polymers
MCLCPs	main chain liquid crystal polymers
Calc.	calculation
4MP4HB	4-methylphenyl 4-hydroxybenzoate
4MP4VEB	4-methylphenyl 4-[2-(vinylloxy)ethoxy]benzoate
4MP4HPB	4-methylphenyl 4-(3-hydroxypropoxy)benzoate
4ABA	4-(allyloxy)benzoic acid
4HBBA	4-(4-hydroxybutoxy)benzoic acid
4ABBA	4-[4-(allyloxy)butoxy]benzoic acid
ppm	part per million
FRIM	Forest Research Institute Malaysia
MPOB	Malaysian Palm Oil Board

LIST OF SYMBOLS

T_g	glass transition state
T_{cl}	clearing point
ν	stretching
δ	bending
vs	very strong
s	strong
m	medium
w	weak

CHAPTER I

INTRODUCTION

1.1 Discovery of Liquid Crystals

Liquid crystals are now well established in basic research as well as in development for application and commercial use. Liquid crystals mean that any of various liquids in which the molecules have partial order and are regularly arrayed in either one dimension or two dimensions, the order giving rise to optical properties such as anisotropic scattering, associated with the crystals. Because they represent a state intermediate between ordinary liquid and three-dimensional solids, the investigation of their physical properties is very complex and makes use of many different tools and techniques.

Liquid crystals play important roles in materials science, as model materials for organic chemists to investigate the connection between chemical structures and physical properties, and providing the insight into certain phenomena of biological systems. Since their main application is in displays, some knowledge of the particulars of display technology is necessary for the complete understanding of the matter (Jonsson, 1992). Friedrich Reinitzer, an Austrian botanist had provided the first scientific description of liquid crystals (Gray, 1998). In 1888, he described his observations of the coloured phenomena occurring in melts of cholesteryl acetate and cholesteryl benzoate. In addition, he noted the “doubled

melting” behaviour in the case of cholesteryl benzoate, whereby the crystals transformed at 145.5 °C into an opaque liquid, which suddenly clarified only on heating to 178.5 °C. Subsequent cooling gave similar colour effects to those observed on cooling the melt of cholesteryl acetate. Reinitzer then approached Otto Lehmann (German scientist) for advice on the optical behaviour of these cholesteryl esters. This led to confirmation of the experiment result of Reinitzer (Brostow, 1992), where Lehmann indicated that the opaque systems observed in Reinitzer studies, were thermodynamically stable states. He also concluded that the structure of these phases had to be intermediate between the crystalline and the fluid state. Lehmann coined for the first time the term ‘liquid crystals’. However Reinitzer was not exactly grateful, he maintained that the name ‘liquid crystals’ is wrong and constitutes a contradiction. Today the coloured phenomena reported by Reinitzer are characteristic of many cholesteric or chiral nematic liquid crystal phases.

Since the publication of Lehmann paper, there was a steady increase in new findings about liquid crystals. Liquid crystal behaviour were also found in purely synthetic materials, such as azoxy ethers prepared by Gattermann and Ritschke (Toyne, 1987) and *p*-metoxycinnamic acid (Jones and Joseph, 1973). But the most significant of which is the work of a group of German scientists leaded by Vorlander who realised that elongated molecular structures (lath-like or rod-like molecules) were particularly suited to mesophase formation (Gray, 1998). His work also showed that if the major axis of a molecule were long enough,

protrusions could be tolerated without sacrifice of the liquid crystal properties. Furthermore he was the first to realise that polymer liquid crystals must also exist. Perhaps the most important consequence of Vorlander's studies was that in laying down the foundation of the relationship between the molecular structure and the liquid crystal properties, attention was focused upon the molecules as the fundamental structural units of the partially ordered phases.

The knowledge base in the fundamental science was also extended greatly by the growth of work on liquid crystal polymers both of main chain and side-chain varieties in 1970s. Amongst others, the names of Percec (1989) and Finkelmann (1987) are associated with the first advances in this field, which attracts many workers today.

1.2 Definitions of Liquid Crystals

Matter exists only either in a solid, liquid or gas forms. Solid may be either crystalline or amorphous. Crystalline solids have a regular arrangement of the molecules over the large distance compared to molecular dimensions. This type of order is called long range order (Slaney *et al.*, 1998). On heating a crystalline solids transform into an isotropic liquid at its melting point. Isotropic liquid does not have a long-range order. Similarly on cooling, isotropic liquid gets converted to crystalline solid. However, there are certain substance like 4-n-pentyl-4'-cyano-bipheyl (PCB) and N-(4-methoxybenzylidene)-4-n-butylaniline (MBBA)

which do not directly pass from crystalline solid to isotropic liquid and vice versa but adopt an intermediate structure which flows like a liquid but still possesses the anisotropic physical properties similar to crystalline solids. Thus, they can be fluid like a liquid and they can have anisotropic properties like crystals. This type of phase is termed **liquid crystal**, liquid crystalline phase, mesophases or mesomorphic phase and the materials are called mesomorphs, liquid crystalline or mesomorphic substances. Nowadays the term liquid crystal, mesomorphic or (mesomorphous) state and mesophase are employed almost synonymously. Substances that under suitable conditions form mesophases are referred to as 'mesogens'.

1.3 Classification of Liquid Crystals

Mesomorphic substances exhibiting liquid crystalline properties in different physical parameters exist either in the presence of a solvent or under a certain temperature intervals. Substances (like sodium or potassium salts of higher fatty acids) that exhibit liquid crystalline phases when dissolved in a controlled amount of solvents are called lyotropic liquid crystals (LLCs). While those substances which exhibit liquid crystalline phases purely due to thermal effects are called thermotropic liquid crystals (TLCs) (Demus, 1990).

1.3.1 Lyotropic Liquid Crystal

LLCs are mainly of interest in biological studies. The most common LLCs systems are those formed by water and amphiphilic molecules i.e molecules that possess a hydrophilic part that interacts strongly with water and a hydrophobic part that is water insoluble such as soaps, detergents and lipids. The most important variable controlling the existence of the liquid crystalline phase is the amount of solvent (or concentration). There are a quite a number of phases observed in such water-amphiphilic systems, as the composition and temperature are varied; some appear as spherical micelles, and others possess ordered structures with one-, two- or three-dimensional position order (Khoo, 1995).

1.3.2 Thermotropic Liquid Crystal

TLCs are formed from compounds (predominantly organic, but also organometalic) either by heating the crystalline solid or by cooling the isotropic liquid. The most widely used and intensively studied TLC systems are for their linear optical properties (Galatola and Oldano, 1998), as well as non-linear optical properties (Marucci and Shen, 1998). The typical molecular structures of TLCs are quite complicated; whose molecules is mainly either rod-, disc-, phasm- (from the name of six-legged stick-like insects), or pyramid-shaped. Depending on the chemical structure and the shape of the constituent molecules or groups of molecules and on external parameters (temperature, pressure, etc.) a rich variety of

mesophases (thermotropic liquid crystalline phases) can be observed (Noel, 1992). There are three main classes of mesophases, namely nematic, cholesteric and smectic. In smectic liquid crystals there are several subclassification in accordance with the position and directional arrangement of molecules. These mesophases are defined and characterised by many physical parameters such as long-and short-range order, orientational distribution functions and so on. TLCs, which are stable at temperatures above the melting point of the compound, are called enantiotropic (Gray, 1987). In certain cases the liquid crystalline phase is only stable at temperatures below the melting point and can be obtained only with decreasing temperature of liquid are called monotropic (Demus, 1990). Presently, most of the interesting applications of liquid crystals have involved those of thermotropic types.

1.4 Liquid Crystal Polymer

Liquid crystal polymer is a new aspect of polymer science. The unusual properties of liquid crystal polymer have led to increasing interest and development in this field. The combination of polymer-specific properties, together with properties specific to the liquid crystal phase has led to a multitude of new prospective which are not possible for conventional materials in the crystalline or amorphous state. However, applications for liquid crystal polymers are still in their infancy. At present only a few materials are commercially available. Consequently, before a material can be applied to a specific problem, a chemical synthesis must be